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TECHNICAL REPORT

AMRA TR 64-19

FRACTURE TOUGHNESS OF D6AC STEEL SHILLELAGH ROCKET MOTOR CASE

by

ROBERT N. KATZ

MATERIALS ENGINEERING DIVISION

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Rocket motor materials

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Fracture toughness

FRACTURE TOUGHNESS OF D6AC STEEL SHILLELAGH ROCKET MOTOR CASE

Technical Report AMRA TR 64-19

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Burst Tests of Shillelagh Rocket Motor Case

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ABSTRACT

Burst tests and fracture toughness measurements on D6AC Shillelagh missile motor cases were carried out. The burst testing was done over a temperature range from room temperature down to -200 F. Results of these tests showed that the motor cases burst at approximately 250,000 psi hoop stress regardless of test temperature, and that all fractures were 100 percent shear. Fracture toughness measurements were made on cases containing machined and fatigue-cracked through-the-thickness notches, at room temperature and -65 F. The results of these measurements were in good agreement with previously obtained flat sheet data on similar materials.

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CONTENTS

INTRODUCTION

The initial design of the Shillelagh missile motor case specified the use of H11 steel. In tests conducted by this Agency during October 1961 on Shillelagh motor cases fabricated from H11 steel of 0.047-inch thickness, it was shown that brittle fracture occurred at -65 F. A recommendation was made to change the material to one which would have high fracture toughness and would behave in a ductile manner at this low temperature. It is interesting to note that subsequent to this recommendation, investigations[1,2] on the fracture toughness of H11 steel have shown that the critical thickness at room temperature for the ductile-brittle transition for material with yield strengths ranging from 190 ksi to about 230 ksi is approximately 0.040 inch. These findings further validate the recommendation to change the material for the Shillelagh missile motor case. The material recommended was D6AC steel.

This report will be concerned with the burst tests and fracture toughness measurements on the Shillelagh missile motor cases. Burst tests of actual Shillelagh motor cases fabricated from D6AC steel were carried out. Values of the fracture toughness calculated directly from motor cases containing through-the-thickness notches were obtained and were correlated with fracture toughness obtained from flat sheet tensile data.[3]

BACKGROUND

To provide technical support for the Shillelagh motor case program, this Agency undertook a project which consisted of a thorough investigation of the smooth and notch tensile properties of 0.040-inch-thick D6AC sheet material.[3] From this investigation it was learned that D6AC steel, which has been austenitized at 1650 F and quenched and tempered in the 1000 to 1100 F range, maintained adequate fracture toughness (K_c greater than 175 ksi $\sqrt{\text{in.}}$) at temperatures well below -65 F, while at the same time maintaining high levels of yield and tensile strengths (yield strength greater than 220 ksi). The relevant smooth and notch tensile data for D6AC steel sheet 0.040-inch thick are presented in Table I.

MATERIAL

The Shillelagh missile motor cases studied in this investigation were provided by the prime contractor at the request of U. S. Army Missile Command. These motor cases were made of D6AC steel forgings whose composition is given in Table II.

TABLE I. Smooth and Notch Tensile Properties of 0.040-Inch D6AC Sheet

Test Temperature (F)	Yield Strength (ksi)	Tensile Strength (ksi)	NTS (ksi)	$\frac{NTS}{UTS}$	Shear (%)	K_{c4} (ksi $\sqrt{in.}$)	$\left(\frac{G_c \text{ in-lb}}{\text{sq in.}}\right)$
1000 F Temper							
RT	216.5	238.5	197.0	0.83	100	182.5	1110
-40	223.5	253.5	193.5	0.77	90	182.5	1110
-120	226.5	256.0	161.0	0.63	85	128.0	545
-160	229.0	262.5	156.0	0.60	40	114.0	435
-200	233.5	271.5	147.5	0.55	25	106.5	380
1050 F Temper							
RT	211.5	237.5	207.0	0.87	100	197.0	1295
-40	215.0	247.5	203.5	0.82	100	192.5	1235
-120	229.0	256.0	169.0	0.66	95	141.0	665
-160	224.5	265.0	154.0	0.58	63	115.5	445
-200	237.5	269.0	143.5	0.53	23	103.0	355
1100 F temper							
RT	209.5	232.0	215.5	0.93	100	207.5	1435
-40	218.5	243.0	205.2	0.85	100	188.0	1180
-120	219.5	244.5	185.5	0.75	90	169.5	960
-160	220.0	251.0	158.0	0.67	95	143.5	685
-200	236.5	264.0	138.5	0.53	20	104.0	360

All specimens austenitized 20 minutes at 1650 F, air cooled.
All specimens tempered 1+1 hour at temperatures indicated.

TABLE II. Chemical Analysis of D6AC Steel

	Elements (weight %)								
	C	Mn	Si	Cr	Ni	P	S	Mo	V
Manufacturer's Analysis	0.43	0.78	0.26	0.97	0.58	0.008	0.009	0.97	0.08
Normal Composition Limits	0.42-0.48	0.60-0.90	0.15-0.30	0.90-1.20	0.40-0.70	0.015 max	0.015 max	0.90-1.10	0.05-0.10

FABRICATION AND HEAT TREATMENT

The Shillelagh missile motor case consisted of three D6AC forgings: one for the gas generator, one for the motor, and one for the bulkhead between these two sections. These forgings ranged in minimum section thickness from 0.040 inch in the gas generator to 0.047 inch in the missile motor and had built-up wall thicknesses next to the bulkheads. The three forgings were electron-beam welded (see Appendix A) to form the complete Shillelagh missile motor case unit. A photograph of the cross section of this motor case is presented in Figure 1. After welding, the motor cases were given the following heat treatment: preheat to 900 F; austenitize in an inert bath for 30 minutes at 1650 F; quench in salt at 400 F for 5 minutes; air cool; temper one hour in salt at 400 to 450 F;

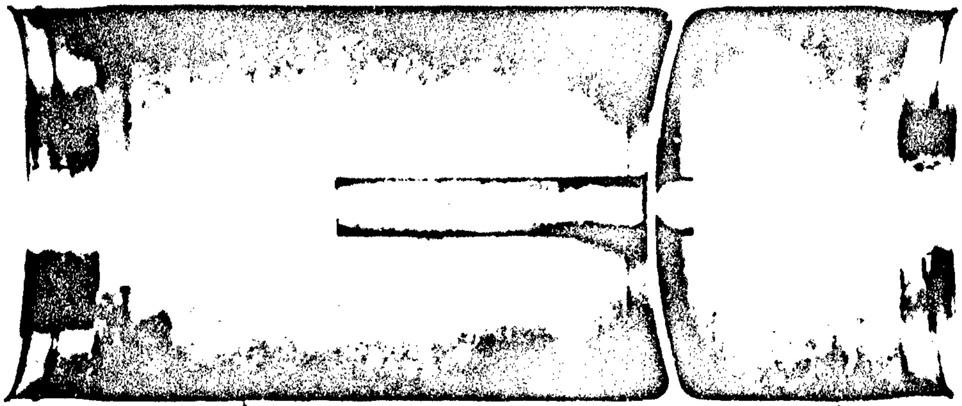

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Figure 1. CROSS-SECTIONAL VIEW OF SHILLELAGH SOLID PROPELLANT MISSILE MOTOR CASE

air cool; and air temper at 1025 F for two hours followed by an air cool. This heat treatment rendered the material fully martensitic with a Rockwell C hardness of approximately 45.

EXPERIMENTAL PROCEDURE

The experiments consisted of evaluating the burst strength of the Shillelagh missile motor cases at temperatures ranging from room temperature down to -200 F, and of measuring the fracture toughness of the cases at room temperature and -65 F. In order to make these measurements, it was necessary to devise pressure packings and end closures which could maintain their integrity at test temperatures as low as -200 F (for details on packings see Appendix B). To measure the fracture toughness, it was necessary to machine through-the-thickness slots in the motor cases. Fatigue cracking of some of the motor cases was also attempted and various means of measuring slow crack growth were investigated.

BURST TESTING

Burst tests were accomplished by packing both ends of the Shillelagh missile motor case, as described in Appendix B and loading hydrostatically by means of a pressurizing fluid. The pressurizing fluids used were water-soluble oil for room temperature tests and ethylene glycol for low-temperature tests. The low-temperature tests were made by immersing the motor case containing the pressurizing fluid in a bath of the desired temperature and allowing the system to equilibrate. Temperature readings were taken by means of thermocouples both within the motor case and in the low-temperature bath. When temperature equilibrium was achieved, the motor cases were loaded to failure.

FRACTURE TOUGHNESS MEASUREMENTS

To calculate the plane stress fracture toughness K_c of D6AC Shillelagh missile motor cases, the following formula[4,5] for the fracture toughness of through-the-thickness notched cylindrical pressure vessels was used:

$$A_{cr} = \frac{1}{\pi}\left(\frac{K_c}{\sigma}\right)^2 - \frac{1}{2\pi}\left(\frac{K_c}{\sigma_{ys}}\right)^2 . \tag{1}$$

Rearranging terms to solve for K_c,

$$K_c = \sqrt{\frac{A_{cr}\pi}{\left[\left(\frac{1}{\sigma}\right)^2 - \frac{1}{2}\left(\frac{1}{\sigma_{ys}}\right)^2\right]}} \tag{2}$$

where

K_c = plane stress fracture toughness
σ = applied stress in vessel normal to crack (hoop stress)
A_{cr} = 1/2 critical crack length (visual estimate)
σ_{ys} = tensile yield strength (at test temperature).

The hoop stress σ was calculated on the basis of the expression:

$$\sigma = \frac{PD}{2t} \tag{3}$$

where

P = burst pressure
D = vessel diameter
t = vessel thickness.

In order to utilize Equation 2 to calculate K_c values for the Shillelagh motor cases, through-the-thickness notches were machined in five of the motor cases. These notches were Elox slots 0.009 inch thick and 0.150 long (one case had a 0.250-inch-long notch). Three of the cases were fatigued to produce natural cracks. Case 110 was cycled from a maximum load of 2000 psi to a minimum load of 200 psi over 375 cycles and between 1500 and 200 psi over 450 cycles, which produced a fatigue crack 0.515 inch long (including the 0.150-inch Elox slot). Case 105 was cycled from maximum loads of 2000 psi to minimum loads at 200 psi over a period of 425 cycles, and at a maximum load of 1500 psi over a period of 173 cycles. This produced a fatigue crack 0.610 inch long. Case 113 was cycled between 2200 and 200 psi 60 times, between 2000 and 200 psi 65 times, and between 1600 and 200 psi 45 times. This resulted in a fatigue crack 0.300 inch long.

In order to maintain the internal pressure in the presence of a through-the-thickness notch, it was necessary to use a patch. The internal patch utilized in this study consisted of a layer of copper next to the notch, backed by a layer of Teflon, which was backed by a layer of Mylar; these layers were approximately 0.010 inch thick. The layers were held in place by neoprene cement.

Several methods of determining the slow crack growth $2A_{cr}$ were tried. One of these was an acoustical method using microphones and vibration-sensitive transducers to pick up the onset of fast fracture. However, the high level of background noise due to the mechanical pumping equipment (used to pressurize the motor cases) precluded the success of this technique. Ink staining also proved unsuccessful due to the ability of the pressurizing fluid to dissolve the stain. The most satisfactory technique proved to be visual determination on the basis of changes in fracture appearance.

RESULTS AND DISCUSSION

The results of the burst tests conducted on the D6AC steel Shillelagh motor cases and fracture toughness measurements on these cases are summarized in Table III.

TABLE III. Burst Test Data for D6AC Shillelagh Motor Cases

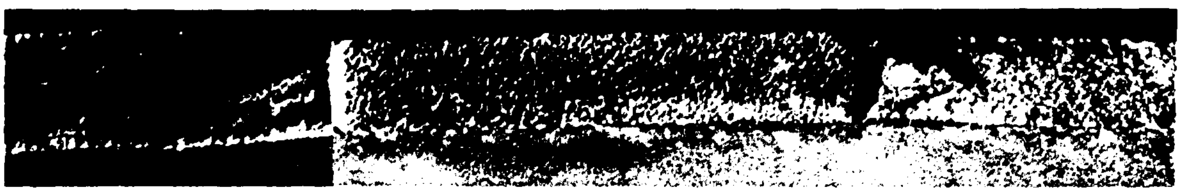
Case	Slot Length (inches)	A_{cr} (inches)	Burst Pressure (ksi)	Hoop Stress σ (ksi)	Approximate K_c (ksi)	Shear (%)	Test Temperature (F)
112	None	-	3.8*	-	-	-	-65
114	None	-	3.7	244	-	100	70
106	None	-	4.0	253	-	100	-55
116	None	-	4.0	254	-	100	-65
111	None	-	3.9	249	-	100	-85
104	None	-	3.9*	-	-	-	-200
115	0.150	0.148	3.1	183	172	100	70
101	0.250	0.300	2.9	183	156	100	-65
110	0.150 fatigued to 0.515	0.257	1.6	102	144**	100	70
105	0.150 fatigued to 0.610	0.305	1.4	88.6	90**	100	70
113	0.150 fatigued to 0.300	0.173	2.4	151	168	100	70

*Case yielded without fracture, end closure leaked.

**Case yielded during fatiguing, hence K_c was low due to strain hardening.

All of the D6AC Shillelagh rocket motor cases broke in the missile motor section of the case, even though the wall thickness in this portion was 0.047 inch as compared to 0.040 inch in the gas generator section. However, the gas generator section had a much smaller length-to-diameter ratio than the rocket motor portion. As a result of this condition, the bulkhead imposed more restraint on the gas generator portion than on the rocket motor portion and therefore failure occurred in the rocket motor portion. Every D6AC Shillelagh motor case which burst exhibited 100 percent shear fracture. This was true from room temperature down to -85 F. The burst hoop stresses which were recorded for the unnotched motor cases were all between 244,000 and 254,000 psi, that is, within 4 percent of each other. It is interesting to note that the two cases which yielded and leaked during testing yielded in this 250,000 psi range. Thus, the D6AC Shillelagh motor case displays an extremely high degree of reproducibility of burst strength even over a wide temperature range.

One case (No. 115) which contained a 0.150-inch-long through-the-thickness notch, but was not fatigue cracked, was tested at 70 F. The K_c calculated from this test was 172 ksi $\sqrt{in.}$, which compares favorably to the value of approximately 190 ksi $\sqrt{in.}$ which may be interpolated from the sheet data presented in Table I. A second Shillelagh motor case (No. 101) containing a 0.250-inch-long through-the-thickness notch with no fatigue crack, was tested at -65 F. This case had a K_c of 156 ksi $\sqrt{in.}$, which, again, when compared with Table I is low but not more than 10 to 15 percent. A photograph of this failed case together with a fractograph of the resultant fracture surface is shown in Figure 2. Two cases



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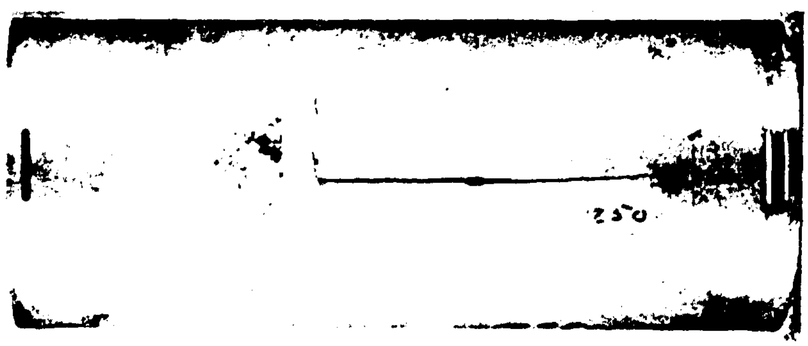
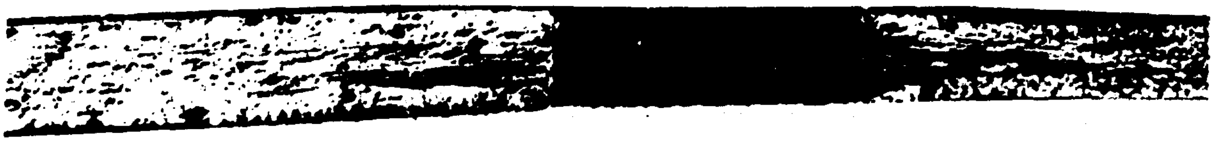
COMPOSITE FRACTOGRAPH SHOWING THE EXTENT OF THE SLOW CRACK GROWTH REGION (BOUNDED BY ARROWS) AND THE FRACTURE SURFACE



FRONTAL VIEW OF BURST MOTOR CASE. THE SHEAR NATURE OF THE FACTURE IS EVIDENT IN THIS PHOTOGRAPH.

Figure 2. CASE NO. 101

(Nos. 110 and 105) were fatigue cracked but were stressed beyond the yield point in the vicinity of the crack during fatiguing,* producing local strain hardening and a resultant change in the fracture toughness in the vicinity of the strain hardening. Thus the values of the fracture toughness parameter K_c obtained from these two cases cannot be considered reliable. However, it is interesting to note that even in these two cases (which, as can be seen from Table III, had the lowest burst strengths and K_c values encountered) the burst pressures were higher than the service operating pressure of 1100 psi. This behavior in the presence of fatigue cracks in excess of 0.50 inch is, to say the least, very desirable. A photograph of Case 110 and its fracture surface are presented in Figure 3.



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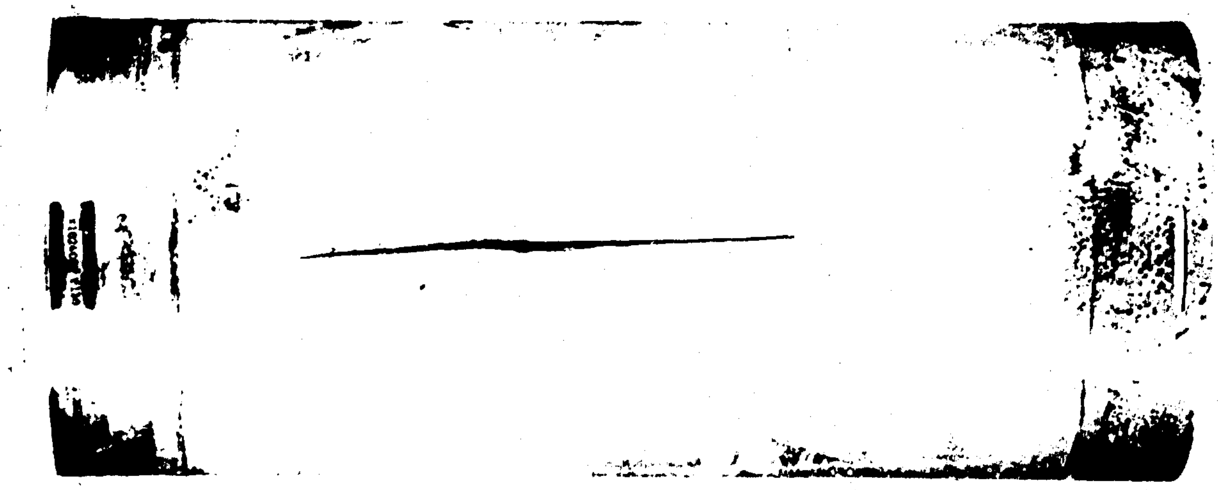
FRACTOGRAPH SHOWING FATIGUE CRACK. NOTE THE DIFFERENCE IN APPEARANCE BETWEEN THIS CRACK INITIATION AREA AND THAT IN THE PRECEEDING FIGURE, NAMELY LACK OF DEFINITIVE SLOW CRACK ZONE AND THE FLATNESS OF FRACTURE.



FRONTAL VIEW OF BURST MOTOR CASE. THE SHEAR NATURE OF THE CRACK (BEYOND THE FATIGUE CRACK) IS AGAIN EVIDENT. SLIGHT YIELDING (BULGING) IN THE NOTCH VICINITY IS ALSO EVIDENT.

Figure 3. CASE NO. 110

*This was only a local phenomena confined to the vicinity of the notch and resulted from the stress concentration due to the notch. The vessel as a whole did not exhibit yielding since the yield strength as determined by the hoop stress formula was not exceeded.

The fatigue cracking of motor case 113 was completed without the incidence of plastic deformation in the vicinity of the crack. The K_c value of 168 ksi $\sqrt{in.}$, obtained by testing this motor case at room temperature, may thus be considered to be reliable. It is indicative of the high degree of reproducibility mentioned above that the room temperature values of K_c for the Elox and fatigue-cracked specimens (Nos. 113 and 115) differ by 4 ksi $\sqrt{in.}$

Although the agreement between the K_c values measured from sheet specimens and those obtained from actual Shillelagh missile motor cases is good, the values obtained from the cases are consistently lower than those measured from the sheet. There may be several reasons for this. First, the K_c values[6] measured from the cases were based on visually estimated values of the slow crack growth region and are therefore equivalent to K_{c1} values,* while the sheet data were based on percent shear measurements and thus were K_{c4} values.* It is known that values based on percent shear measurements are generally higher than those based on slow crack growth measurements. Secondly, the possibility exists that bulging of the internal patch could act as a wedge at the inner surface of the notch and thus reduce the measured fracture toughness. Of course, even in the absence of the above effects, the differences in heat treatment and processing history between the sheet material and the actual motor cases could account for the observed differences in fracture toughness.

CONCLUSIONS

1. The D6AC steel is more reliable than H11 steel for use in the Shillelagh missile motor case because of superior fracture toughness at low temperatures.

2. The D6AC Shillelagh motor cases tested evidence an extremely high degree of uniformity and reproducibility of properties.

3. Measurements of the fracture toughness may be made from actual motor cases using visually estimated slow crack lengths. These measurements are in good agreement with sheet data previously obtained for material of the same thickness and heat treatment.

4. The Shillelagh motor case fabricated from D6AC steel can have a through-the-thickness crack greater than 1/2 inch long and still tolerate pressures exceeding the design pressure.

*K_{c1} refers to the fracture toughness parameter using a center notch, and measuring the slow crack length. K_{c4} refers to the fracture toughness parameter using edge-notched specimens and a correction on term based on the percent shear which corrects for the plastic zone ahead of the crack.

ACKNOWLEDGMENTS

The author would like to express his appreciation to Mr. Edward LeMay of the AMRA High Pressure Facility for his assistance in conducting the burst testing of the Shillelagh motor cases, and to Mr. Kenneth Abbott, Chief of the Weapons Materials Section, for his help at all stages of this study, but especially in the redesign of the low-temperature pressure packings.

APPENDIX A

ELECTRON BEAM WELDS

Since the fabrication of the Shillelagh missile motor cases included electron-beam welding, it would be appropriate to discuss briefly the behavior of the electron-beam welds. Historically, welds have been the "Achilles' heel" of many solid propellant missile motor cases. It is interesting to note in the tests this Agency performed on the Shillelagh missile motor cases that:

a. no cracks or failures initiated in the welds; and

b. cracks, when running through the welds, would not propagate either along the weld or in the heat-affected zone.

Figure A-1 shows an early electron-beam weld on D6AC material. A clearly defined weldment and heat-affected zone can be seen. Figures A-2 and A-3 show welds cut out of Shillelagh motor cases 115 and 106. These weld segments were taken from the motor case in the vicinity of the crack. It is observed that the weldment and heat-affected zone are almost indistinguishable microstructurally from the matrix material and that the fracture is in the base metal, not the heat-affected zone. Similar microstructural observations were made by Kern and Lubin[7] for electron-beam welded D6AC heat treated after welding (as was the Shillelagh motor case).

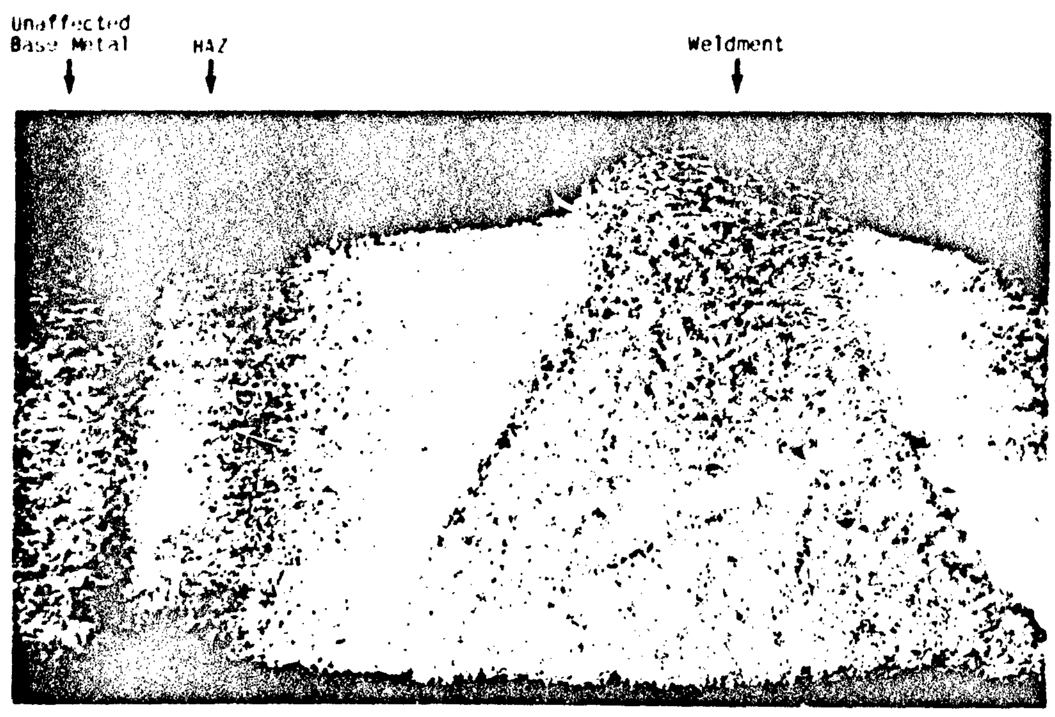


Figure A-1. EXAMPLE OF EARLY ELECTRON-BEAM WELD IN D6AC SHEET

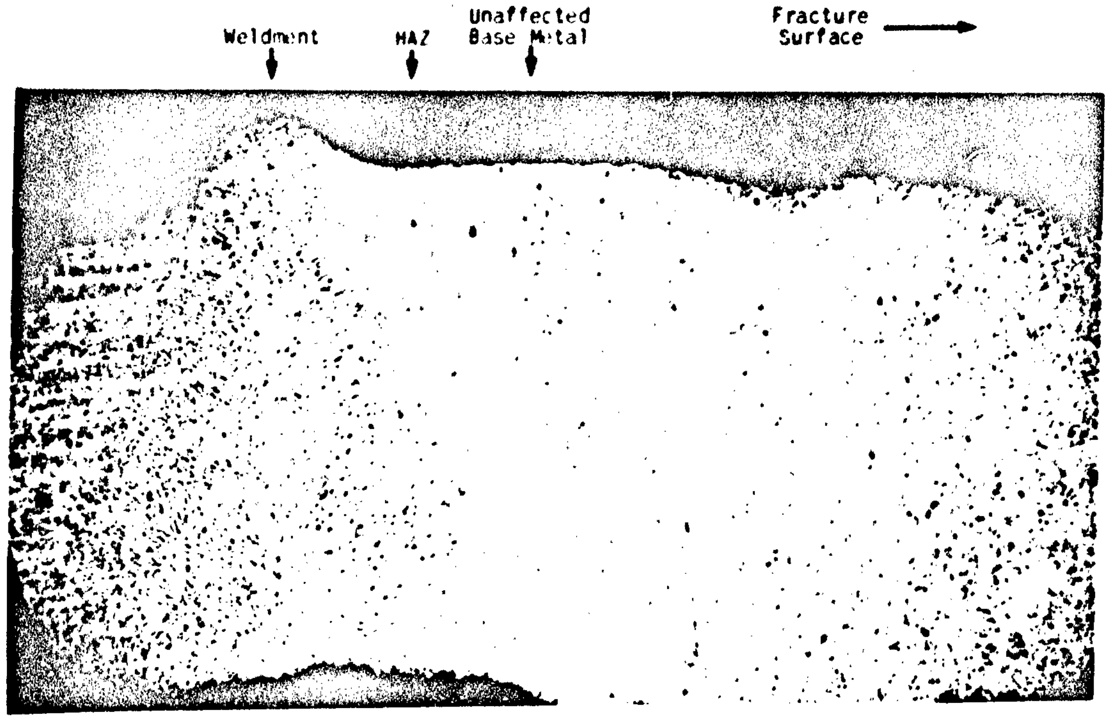


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Figure A-2. ELECTRON-BEAM WELD IN CASE 115

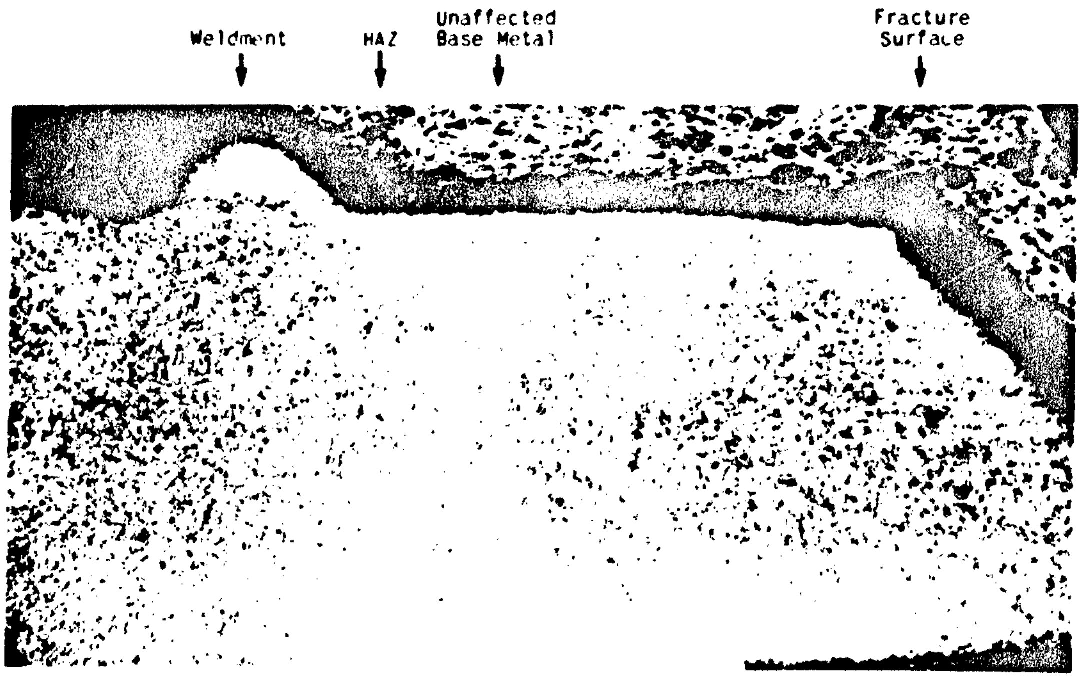

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Figure A-3. ELECTRON-BEAM WELD IN CASE 106

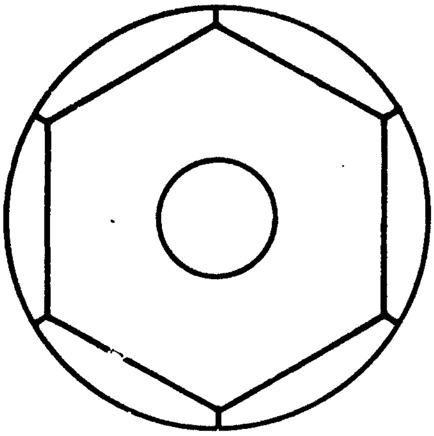
APPENDIX B

END CLOSURE AND PRESSURE PACKING DESIGN

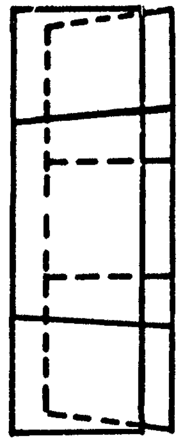
The end closure and pressure packing design which was chosen for use at low temperatures (also used at room temperature) consisted of a top packing support, packing, and bottom packing support. The top packing support was comprised of a split-ring packing follower seated on a solid packing restrainer. The packing follower split ring was made of segments which, when in place, formed a circle with their outer faces and a concave hexagon with their inner faces. This concave hexagon fit over a convex hexagonal surface on the packing restrainer. The hexagonal design of the contact surfaces between packing follower and packing restrainer allowed some individual motion of each segment of the follower without segment cocking which would result in a leak.

Upon the application of pressure the packing restrainer is forced upward. This upward motion of the packing restrainer results in radial motion of the packing follower ring segments. To prevent yielding of the motor case as a result of the radial movement of the packing follower segments, it was necessary to use heavy restraining rings around the motor case ends.

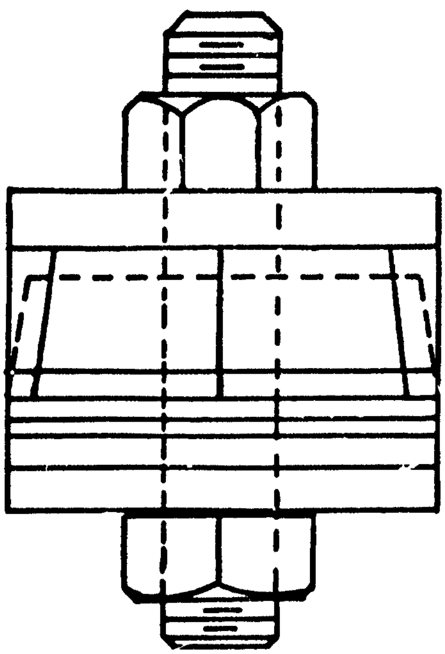
The packing consisted of a layer of Thiokol-impregnated leather, approximately 1/8-inch thick; a layer of silicon rubber, approximately 3/8-inch thick (this material has a glass transition temperature of about -110 C, which allows it to maintain its rubber elasticity at temperatures well below other rubbery polymers); and a layer of Teflon, approximately 1/8-inch thick. This packing, with top restrainer and follower on top of it, was supported by the bottom restrainer which consisted of a simple disk. The entire assembly was held together by a rod which was locked with a nut at either end of the packing. On one packing this rod contained a center bore allowing admission of pressurizing fluid to the motor case. The end closure assembly is shown in Figure B-1. After insertion into the case, the restraining ring was placed around the case in the vicinity of the packing.



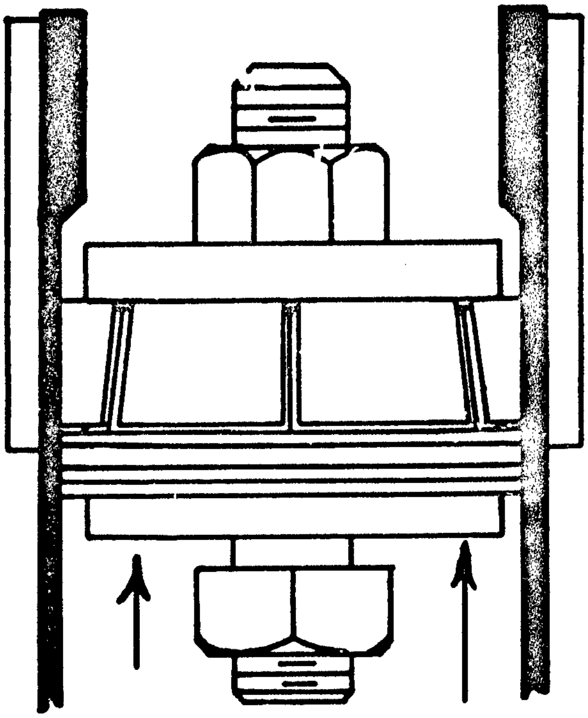
TOP VIEW OF TOP PACKING SUPPORT
RESTRAINER AND FOLLOWER



SIDE VIEW OF TOP
PACKING SUPPORT



CROSS-SECTIONAL VIEW OF
ASSEMBLED PACKING



CROSS-SECTIONAL VIEW OF PACKING IN MOTOR
CASE WITH RESTRAINING RING IN PLACE

Figure 3-1

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Burst tests and fracture toughness measurements on D6AC Shillelagh missile motor cases were carried out. The burst testing was done over a temperature range from room temperature down to -200 F. Results of these tests showed that the motor cases burst at approximately 250,000 psi hoop stress regardless of test temperature, and that all fractures were 100 percent shear. Fracture toughness measurements were made on cases containing machined and fatigue-cracked through-the-thickness notches, at room temperature and -65 F. The results of these measurements were in good agreement with previously obtained flat sheet data on similar materials.

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